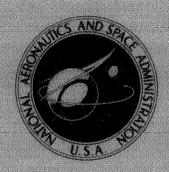
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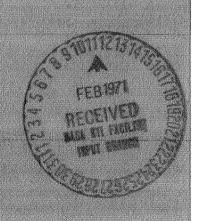


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APPLICATION OF SERT II THERMAL CONTROL COATINGS

by N. John Stevens Lewis Research Center Cleveland, Ohio 44135



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APPLICATION OF SERT II THERMAL CONTROL COATINGS

by N. John Stevens

Lewis Research Center

SUMMARY

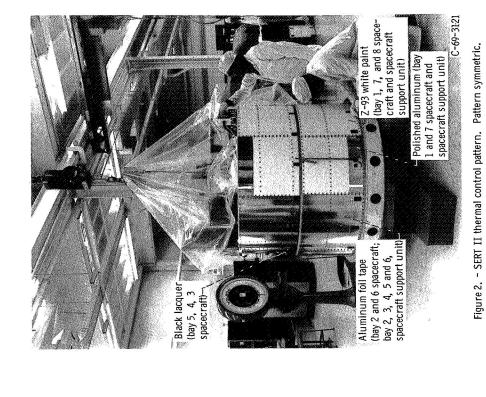
The coatings required for the passive SERT II thermal control system are a white inorganic paint (Z-93), aluminum foil tape, and polished aluminum. While information on the performance of these coatings could easily be found, the detailed procedures for applying these coatings are lacking.

In this report, the development of the application techniques for these coatings is presented. The factors that influence the adhesion of the white Z-93 paint are discussed. A solution to the problem of blistering or lifting of tapes is given. A general discussion of the polishing procedure is included. The testing program performed on the coatings to demonstrate their stability is also presented.

INTRODUCTION

The purpose of the second Space Electric Rocket Test (SERT II) mission is a long-term life test of a 1-kilowatt mercury bombardment ion thruster in a space environment. The satellite was placed in a 540 nautical mile, circular, polar orbit on February 3, 1970. It is oriented such that one of the cylindrical sides will be in constant sunlight for at least 10 months. In its orbiting configuration, the SERT II satellite consists of the Agena D vehicle with the solar array attached, a spacecraft support unit (SSU) which houses the command system, telemetry, and control moment gyros (CMG), and a spacecraft section which houses the two ion thrusters and the associated experiments (see fig. 1). The details of the SERT II mission and experiments can be found in the literature (refs. 1 to 4).

The thermal control of this satellite was to be accomplished by passive means using existing thermal control coatings. Since the Agena section was to be dormant after placing the satellite in its proper orbit configuration, it was decided that its thermal control system should be dictated by the ascent constraints rather than orbiting consideration. Hence, the thermal design was narrowed to finding coatings for the operating portions



Agena D --

Solar array-

--- Spacecraft support unit

Spacecraft fon thruster

- Forward equipment rack

Figure 1. - SERT II satellite.

Earth

of the satellite, the spacecraft section and the SSU. The coatings chosen for the thermal control system were zinc oxide - potassium silicate inorganic white paint known as Z-93, aluminum foil tape, polished aluminum, and black paint (see fig. 2). The optical properties of these coatings under a variety of environmental conditions could be found in the literature (refs. 4 to 19). However, what is not discussed is the detailed procedures involved in applying these coatings.

This report describes the method of application of the coatings used in the SERT II program, the problems encountered, and the solutions that were devised. Since the black paint application did not create any unique problems, it is not discussed further in this report. The other three coatings are discussed in separate sections. Each section consists of background information, a detailed discussion of the application technique, the evaluation testing to which the coating was subjected, and a summary.

APPLICATION OF ZINC OXIDE - POTASSIUM SILICATE WHITE PAINT

Background

Early in the thermal design effort for the SERT II vehicle, it was realized that a stable, low absorptance - high emittance coating would be necessary to satisfy the constraint of using a passive thermal control system. Approximately 20 square feet (1.86 m²) of surface area on six of the Sun facing panels had to be coated with this solar reflector coating. The information available in the literature indicated that the zinc oxide - potassium silicate white paint known as Z-93 would satisfy this requirement (refs. 4, 6, and 15 to 17). However, in common with other silicate paints, the Z-93 paint was reputed to be difficult to apply and its rough porous surface had the characteristic of being easily contaminated by dirt, oils, fingerprints, lint, and tool marks (ref. 20).

The initial efforts in working with the Z-93 paint on the SERT project did not confirm its reputation. The paint was prepared according to the published procedure (ref. 6). The aluminum and magnesium substrates were cleaned by a vigorous sanding followed by a water wash. The actual painting with a hand-held spray gun was accomplished with little difficulty. Optimum paint thicknesses of 5 mils $(1.3\times10^{-4} \text{ m})$ were applied (ref. 6). The air-cured coating appeared to adher well to both of the substrate materials, withstand thermal testing, and possess the desired optical properties. When the work progressed to the prototype stage, however, difficulties with the paint adhesion appeared. The paint applied to the magnesium prototype panels started to flake off the panels about 1 week after application (see fig. 3). This peeling or flaking off of the paint was more pronounced when the panels were exposed to a thermal-vacuum environment (see fig. 4).

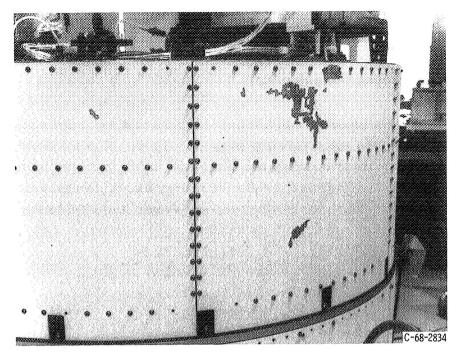


Figure 3. - Self-lifting of Z-93 paint from magnesium in air.

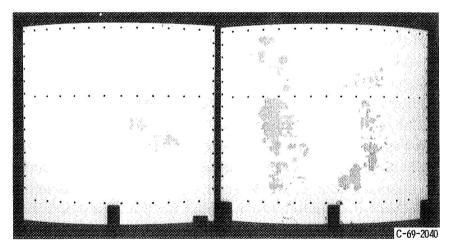


Figure 4. - Loss of adhesion of Z-93 on magnesium panels after thermal vacuum testing.

This problem necessitated a more thorough investigation of the painting process. The first adjustment made was to change the panel material to aluminum as recommended by Illinois Institute of Technology Research Institute (IITRI), the developers of the paint. The choice of aluminum alloy was to be determined as part of the application study program.

Application Studies

After the prototype panel failures, a program was undertaken to determine how various parameters affected the reliability of the Z-93 paint application. The parameters selected for study were the calcining or heat treatment of the zinc oxide powder, paint cure requirements, substrate material, cleaning techniques, and environmental effects. The 4-inch (0.1-m) square samples used in these tests were subjected to a bend adhesion test, thermal shock, and optical property evaluation. Full-size SERT vehicle panels (about 2 ft (0.6 m) square) were also painted as practice for the painter and were subjected to thermal-vacuum testing. The aluminum surfaces were protected by an Irridite 14.2 conversion coating (MIL. Spec. C-5541A) which was removed in the sanding operation. It was decided that the paint would be ball-milled according to the standard methods (refs. 6 and 21) and that the paint would be applied by hand-held pressure spray gun. Work was also done with an automatic spray paint machine, but problems with variable spray gun pressures and paint curing soon caused the termination of this effort. This work with the paint machine is discussed in appendix A of this report. The detailed process specification that resulted from these studies is given in appendix B.

Zinc oxide calcining and paint cure investigation. - The question of the proper calcining or heat treatment of the zinc oxide powder appeared to be an unresolved one. The original work with the Z-93 paint (ref. 6) stated that the zinc oxide must be calcined to obtain a stable pigment; the calcining procedure used was to heat the powder in air at 700° C (1300° F) for 16 hours. Yet, in the Z-93 paint specification (ref. 21), the calcining temperature is given as 1100° to 1300° F (600° to 700° C). Furthermore, the specification stated that air curing was acceptable but that a bakeout in the cure cycle would improve the adhesion of Z-93 paint. A series of tests were undertaken to investigate the effect of the calcining temperature and the bakeout curing on the paint adhesion.

It was first learned by trial that calcination of the powder is not only required for a stable pigment but also to make a thin, sprayable paint. With the uncalcined powder, the paint mixture is too viscous to spray. For this calcining temperature investigation, zinc oxide from the same batch of SP-500 powder purchased from New Jersey Zinc Company was calcined in three 5-pound (2.3-kg) groups: one each at 1100° , 1200° , and 1300° F (600° , 650° , and 700° C). The powder was ball-milled with the proper amounts of po-

tassium silicate and distilled water to make the three batches of Z-93 paint (see appendix B). The paint formulated from one of the calcined powders was applied the same day that it was mixed to a group of six 4-inch (0.1-m) square test specimens of Alclad 2024. The surface preparation of the Alclad consisted of a wet sanding with wet or dry paper followed by a distilled water rinse. The painting procedure was to apply the first wet coat of paint consisting of two spray passes at right angles to each other, then bake two of the six samples in an oven at 270° F (130° C) for 1/2 hour, while the other four were allowed to air cure. Then, the second wet coat was applied about 2 hours later to all six of the samples. After this second coating, the two samples that were baked and two of the other four samples were subjected to a bakeout at 270° F (130° C) for 2 hours. Hence, the cure cycles were air cure only (2 samples), a single 2-hour bakeout after the second coating operation (2 samples), and bakeouts after the first and second coating operation (2 samples). This procedure was repeated for each of the paints made from the other calcined powders for a total of 18 samples (see table I). The paint thick-

TABLE I. - SUMMARY OF Z-93 PAINT CALCINATION INVESTIGATION

| Sample | Calcination | Paint cure | | | Sample use | | |
|--------|---|------------|-------------------|------------------|---------------------------|--|--|
| | tempera- ture, ^O F (^O C) | Air | Single bakeout | Two bake- out | | | |
| 1 | 1100 (600) | х | | | Bend adhesion | | |
| 2 | | X | | | Optical and thermal shock | | |
| 3 | | - | Х | | Bend adhesion | | |
| 4 | | | х | | Optical and thermal shock | | |
| 5 | | | | Х | Bend adhesion | | |
| 6 | | | | х | Optical and thermal shock | | |
| 7 | 1200 (650) | х | | | Bend adhesion | | |
| 8 | | X | | | Optical and thermal shock | | |
| 9 | | | х | | Bend adhesion | | |
| 10 | | | x | | Optical and thermal shock | | |
| 11 | | | | X | Bend adhesion | | |
| 12 | | | | X | Optical and thermal shock | | |
| 13 | 1300 (700) | x | |) Mail dans | Bend adhesion | | |
| 14 | | x | | | Optical and thermal shock | | |
| 15 | | | Х | | Bend adhesion | | |
| 16 | | | x | | Optical and thermal shock | | |
| 17 | | | | х | Bend adhesion | | |
| 18 | | | | х | Optical and thermal shock | | |

ness on all of the samples was approximately 5 mils (1. 3×10^{-4} m) as measured with an eddy current meter.

The adhesion of the paint to the substrate was checked by the bend adhesion test. This test consists of placing a sample in a conical mandrel bend tester, bending it 180° , removing it from the apparatus, placing a strip of 3M no. 250 tape on the bent area and pulling it off. If more than 20 percent of the paint in the bent area came off, the sample was considered to have failed the test. This test imposes conditions that are more severe than any expected flight conditions, and guarantees, therefore, that the paint will

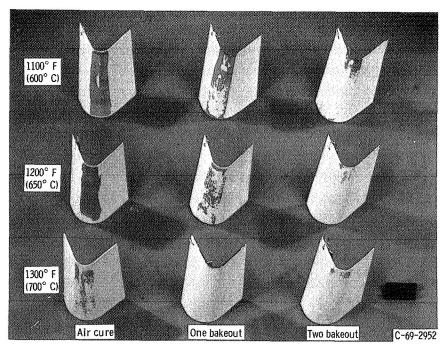


Figure 5. - Bend adhesion test results (calcining study).

adhere to the surface. In figure 5 the results of this test are shown. The specimens are arranged in rows for the three calcining temperatures, and in columns for the different cure procedures. The adhesion of the air-cured samples is shown to be improved with the higher calcining temperatures. The bakeouts do improve the adhesion of the paint, and the double bakeout cure is better than the single bakeout in promoting adhesion.

A second test of the mechanical properties was the thermal shock test. This consisted of heating a sample to 490° F (250° C) and then plunging the hot sample into a liquid nitrogen bath. This was followed by another heating-cooling cycle. All of the samples passed this test. No cracks were visible even when viewed under 20 power magnification.

In conducting this test it was found that the 1300° F (700° C) calcined powder could be mixed in the standard proportions to obtain a sprayable paint. Paint made from the lower temperature calcined powder required more thinning with water to make the mixture sprayable. Since the original work with Z-93 paint was conducted using 1300° F (700° C) calcined powder, it was suspected that the paint mixture should have been revised to account for the powder properties at various calcining temperatures. No further work was done on this point, and the standard procedure of thinning with distilled water, as required, was adopted. A viscosity of between 65 and 68 Krebs units, as measured by a Thomas Stormer Viscosimeter, was used as a guide. (Krebs units are a measure of the time it will take a driving weight to produce 100 revolutions of a paddle wheel in the paint. For example, 65 to 68 Krebs units corresponds to 28 to 31 sec for a 125-g driving weight to produce the 100 revolutions.)

The spectral reflectance curves of samples painted in this test series were obtained using a Cary 14 Spectrophotometer. These curves are presented in figure 6. Values of solar absorptance can be calculated from these reflectance curves (ref. 5) using Johnson's solar intensity curve. For the range of wavelengths of 0.25 to 2.5 microns, the computed absorptances are 0.14 for 1100° F (600° C) calcined powder, 0.15 for the 1200° F (650° C) powder, and 0.17 for the 1300° F (700° C) powder.

This phase of the program has shown that both the calcining temperature of the zinc oxide powder and the baking of the paint have a pronounced effect on the adhesion of the Z-93 paint. The higher calcining temperatures change the zinc oxide powder such that the adhesion of paint made from this powder is improved. This change, however, shifts the solar spectrum response which results in a paint that has a higher solar absorptance. The limit on the calcining temperature is reached when the zinc oxide powder is

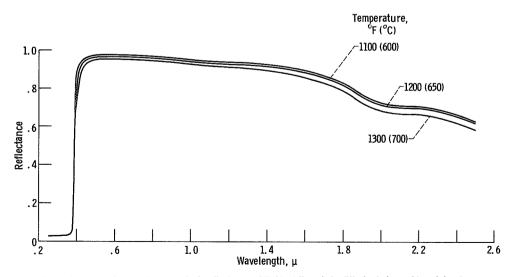


Figure 6. - Room temperature spectral reflectance of Z-93 white paint. Effect of zinc oxide calcine temperature; Cary 14 spectrophotometer.

permanently yellowed. This will occur above 1300° F (700° C). At calcining temperatures around 1100° F (600° C), the paint is viscous, difficult to spray, and has poor adhesion, even with bakeouts added to the cure cycle. The addition of bakeouts to the cure cycle of Z-93 paints made from 1200° and 1300° F (650° and 700° C) powder definitely improves the adhesion without changing the optical properties.

Substrate evaluation. - A series of tests were run to evaluate various substrates and various methods of surface preparation. The materials used were 4-inch (0.1-m) squares of Alclad 2024, 6061 aluminum, and 2024 aluminum. The surfaces to be painted were prepared in three different ways: sanding with 220 grit wet or dry paper, vaporhoning, or glass beading. After surface preparation, the pieces were washed in Alconox detergent and rinsed with distilled water. The Z-93 paint made from 1200° F $(650^{\circ}$ C) calcined powder was sprayed on all of the samples at the same time to nominal thickness of 5 mils $(1.3\times10^{-4}$ m) in two coating runs. The paint adhesion as determined by bend adhesion tests was found to be independent of the type of aluminum substrate and the surface preparation used in this series of tests.

Cleaning techniques. - After the calcining investigation was completed, the rubprime technique for improving the Z-93 paint adhesion was incorporated into the application procedure. This procedure was suggested by IITRI, who acted as consultants on the Z-93 paint application problems. It consisted of thoroughly cleaning the substrate by sanding followed by a detergent (such as Alconox) and cleanser (tri-sodium phosphate or equivalent) scrubbing until the substrate was break-water free. This means that a flow of water over the surface will show no evidence of breaking or dividing into separate water flows. Hence, the surface was assumed to be clean. Then, a light coat of Z-93 paint was firmly hand-rubbed into this clean surface with cheesecloth. Once this prime coat had dried, it should not come off when flushed with water or when rubbed lightly with a wet cloth. If the rub prime coat does disappear, the surface is not clean enough, and the cleaning procedure must be repeated. The wet coat application was made by first mist coating the Z-93 prime coat with distilled water (until the surface has a wet water gloss) and then painting to the desired thickness. With this technique added to the Z-93 paint process, excellent adhesion could be obtained with 1200° F (650°C) calcined powder, provided the environmental conditions were conducive to good drying of the paint.

Environmental conditions. - In the painting program a common problem was the varying surface texture that was obtained even though the paint preparation and application were supposed to be identical. The desired application technique was one that would allow the paint to be applied as a continuous, wet film yet dry to the rough textured surface typical of Z-93 paint. At times, the 1200° F (650° C) calcined powder would mix up into a thin paint (viscosity of 65 Krebs units) without any additional water thinning required. This paint would dry to a glossy smooth coating that looked excep-

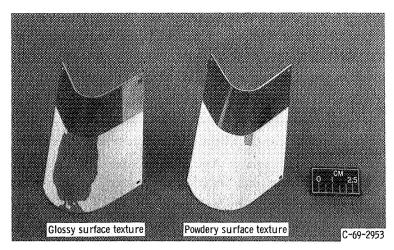


Figure 7. - Bend adhesion test results for effect of surface texture.

tionally good but had very poor adhesion (see fig. 7) even with the rub-prime technique included. It appeared that the 1200° F (650° C) calcined powder mixed up into a paint that had the viscosity of 1300° F (700° C) calcined powder paint and resulted in a paint that had the adhesion typical of a 1000° F (540° C) calcined powder paint. The optical properties, however, were the same for both the smooth surface paint and the rough.

A solution to this problem was found when it was discovered that the smooth glossy surface usually developed during periods of high relative humidity (60 to 80 percent). When the relative humidity was less than 55 percent, the paint would dry to the desired rough surface texture that had excellent adhesion. Hence, it was deduced that the problem was related to the drying of the paint: in the humid conditions the water could not evaporate. The solution was to bakeout the panels immediately after the wet coats were applied. This quick-bake of the panels was accomplished by holding them under a lamp bank after the first wet coat application until the surface steam stopped rising. At this time the paint developed the rough surface texture desired. The panels were hand held under the lamp bank to avoid the necessity of removing the masking on the panels. The panels were allowed to cool, usually from 1 to 4 hours, then mist-coated with distilled water, and finally the second wet coat was applied. A second quick-bake under the lamp bank followed the second wet coat application. Then the panels were left to air cure overnight. The demasking and final oven bakeout followed the next day.

Evaluation

The Z-93 paint applied to the SERT II panels had to be evaluated to determine the optical and mechanical properties. This was especially important since the evaluation

philosophy was that a flight test set of panels were to undergo all environmental testing with the flight vehicle, while the actual flight panels would be added to the satellite only just prior to final shroud installation. Furthermore, no optical measurements were to be made on the flight panels themselves. These flight panels were to be painted with the same processing as the flight test panels but were only subjected to a thermal-vacuum test in an ion-pumped facility. All optical and mechanical evaluations were to be made on 4-inch (0.1-m) square samples painted at the same time as the flight panels. These tests were in addition to the bend test and thermal shock test required by the paint process specification.

Optical properties. - The solar absorptance of the Z-93 paint was determined from room temperature reflectance measurements using the Cary 14 spectrophotometer, from vacuum calorimetric measurements (ref. 22), and from Lion Optical Comparator measurements. The emittance of the Z-93 paint was determined from the vacuum calorimetric measurements (ref. 22) and from the Lion Optical Comparator readings. The measured solar absorptance of the Z-93 paint by the Cary 14 spectrophotometer is shown in figure 8 as a function of thickness of the paint. The curve was generated from data given in reference 6. The agreement between the SERT data and this reference is excellent, which indicates that the SERT procedure results in a paint that is as good optically as could be expected. The optical property measurements are shown in table II along with the design values.

The Lion instrument was used to make periodic checks on the coatings to determine if there was any contamination of the Z-93 paint in testing or storage. These tests were made on 4-inch (0.1-m) square test specimens that accompanied the full-size panels from paint application to launch.

Environmental testing. - All panels coated with Z-93 paint were required to be subjected to a week of thermal-vacuum testing cycling between temperatures of 140° and -40° F (60° and -40° C). The panels were held at the temperature extremes for a minimum of 48 hours as well as cycling five times between the extremes. This was generally more than enough time for evidence of poor adhesion to show up. If the paint had been applied well, then the panels would come out of this thermal-vacuum test looking like those shown in figure 9.

Solar simulation. - The optical stability of the coatings exposed to sunlight in flight was checked by running a solar simulation test for 400 hours. This test was conducted on 4-inch (0.1-m) square samples of Z-93 paint and polished aluminum. A 1-inch (0.025-m) square optical solar reflector was used in the test to monitor the constancy of the simulator beam.

The samples were suspended by nylon thread in a vacuum chamber such that they would be illuminated by the solar simulator beam which entered the chamber through a quartz port in the tank wall. The simulator was an Aerospace Control solar simulator

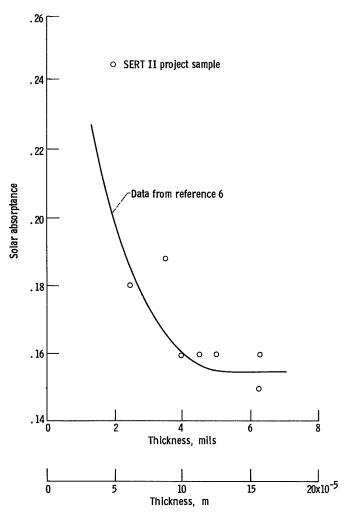


Figure 8. - Variation of solar absorptance with thickness for Z-93.

TABLE II. - OPTICAL PROPERTIES OF SERT II COATINGS

| | Solar absorptance, $ lpha_{_{f S}} $ | | | | Emittance, ϵ | | | |
|---|--------------------------------------|----------------------------|-----------------------|--------------------------------------|----------------------------|-----------------------|-----------------------------------|--|
| | Cary 14 | Vacuum calori- meter | Lion | Design value | Vacuum calori- meter | Lion | Design value | |
| Z-93 white Aluminum foil tape Polished aluminum | 0. 16 . 13 . 26 | 0. 18 . 14 | 0. 19 . 19 . 28 | 0. 16±0.03 . 14±0.03 . 26±0.05 | 0.92 .07 | 0. 95 . 03 . 04 | 0.90±0.05 .06±0.03 .06±0.03 | |

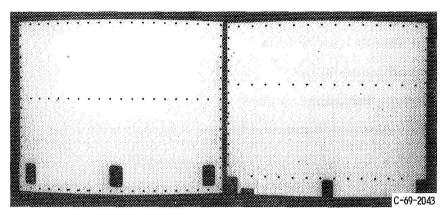


Figure 9. - Z-93 painted panels after thermal vacuum testing.

which used a filtered xenon lamp system. The intensity and spectral distribution of the beam were measured at the test plane through the quartz window to determine the test conditions. At the two solar constant intensity used in this test, the ultraviolet content was about $1\frac{1}{2}$ times the value in the solar spectrum. Therefore, the 400 hours of testing would result in approximately 600 hours of solar ultraviolet exposure. This was considered to be sufficient time to determine if the coating was stable.

A single thermocouple was placed on the rear side of each sample, which was painted black. After the vacuum tank pressure was reduced to less than 10^{-5} torr, the simulator was turned on and the shroud temperature was adjusted to give an initial Z-93 paint sample temperature of about 90° F (32° C). All sample temperatures and shroud temperatures were continuously recorded on strip chart recorders. The tank pressure was at or lower than 10^{-7} torr throughout the 400 hours of testing.

The value of the absorptance of the sample for the lamp spectrum can be computed from the following approximate energy balance:

$$I\alpha A_1 = \sigma \left[\epsilon_B A_B \left(T^4 - T_s^4 \right) + \epsilon_F A_F \left(T^4 - T_s^4 \right) \right]$$

or

$$\alpha = \frac{\sigma}{I} \left[(\epsilon_{\rm B} + \epsilon_{\rm F}) \left({\rm T}^4 - {\rm T}_{\rm s}^4 \right) \right]$$

where

 $A_1 = A_B = A_F$ area of one side of test sample ϵ_B , ϵ_F emittance of back and front sides

I intensity of light at test plane

T test sample temperature T_S shroud temperature σ Stephan-Boltzmann constant σ absorptance of the sample for the lamp spectrum

The view factors were set equal to one for this calculation. The emittance of the back side of the sample $\epsilon_{\rm B}$ had been measured, and the emittance of the front side $\epsilon_{\rm F}$ was assumed to be constant at the initial value.

The sample temperatures are shown in figure 10(a) while the computed values of absorptance are shown in figure 10(b). The increasing sample temperatures toward the end of the test are caused by an increase in the shroud temperature. Hence, the absorptance computed from the previous formula results in a constant value. The constancy of the computed value of absorptance over the testing period is taken as strong evidence that the SERT II paint application process does not introduce contamination into the coating. The computed absorptance value of 0.16 to 0.17 also agrees well with the absorptance values obtained from the Cary 14 spectrophotometer data. This indicates that the lamp spectrum does approximate the solar.

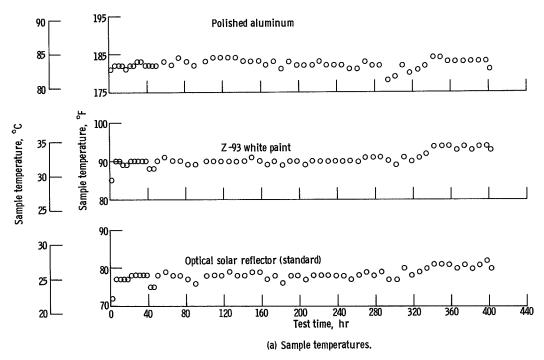
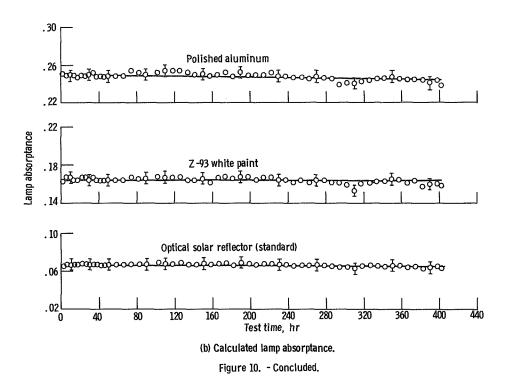


Figure 10. - Solar simulation test.



Summary Remarks

In order to apply Z-93 paint successfully, there are guidelines that must be followed. First, the substrate must be cleaned and kept clean until the paint is applied. The rub prime technique offers a good way in which the cleanliness can be verified before the surface is painted. Once a surface is well primed in this manner, the surface can be left overnight for Z-93 paint application on the following day. If the surface cleanliness is poor, then the paint applied to these surfaces cracks and peels off in air, usually within a week. The aluminum alloy used does not influence the adhesion of the paint.

Second, the paint must be prepared properly. The zinc oxide powder must be heat treated or calcined in air to make it into a useable thermal control paint. Calcining the powder at or below 1100° F (600° C) results in a paint that is viscous, is difficult to spray, and has very poor adhesion. Calcining above 1300° F (700° C) results in a permanent yellowing of the powder and a paint that has a higher solar absorptance than desired. In the SERT II project the zinc oxide was calcined at 1200° F (650° C) for 16 hours. There was no advantage in going to the higher temperature since the final application procedure for the paint made from the 1200° F (650° C) calcined powder resulted in a coating that was just as adherent. The ratio of the zinc oxide to the potassium silicate must be maintained, but there is some question about the quantity of distilled water that should be used. Since the water is just the thinner, not a chemical

constituent of the paint, more water can be added after the paint is mixed to make paint sprayable. For the SERT II project, the Thomas-Stormer viscosimeter was used to ensure that the paint viscosity was between 65 and 68 Krebs units.

Third, the paint application can vary with local environmental conditions. When spraying in periods of low humidity, the paint will dry to the desired rough surface texture by itself. In periods of high humidity, the painted surface appears glossy when wet and will dry to a smooth, glossy surface which will exhibit poor adhesion. This latter condition can be avoided by adding a bakeout after each wet coat to drive out the water. If the quick bake procedure is used, then the bakeout should be terminated when the steam stops rising from the surface.

Finally, the problem of protecting the applied Z-93 paint must be considered for the paint is easily contaminated in the handling, testing, and storage of the finished product. In the SERT II project, this problem was alleviated in that the flight panels were removable and could be replaced at the launch site. Hence, the flight panels were painted, handled with clean nylon gloves, tested, wrapped in clean kraft paper, and sealed in their shipping container. Even with these conditions, test samples were constantly used to verify that the optical properties remained at the original value until launch.

APPLICATION OF ALUMINUM FOIL TAPE

Background

The pressure-sensitive aluminum foil tape (Mystik no. 7402L) used in the SERT II thermal control pattern as a low emittance coating was reputed to provide a relatively simple thermal control coating application. The tape had been used previously in space flight (ref. 23). The problems encountered in its application have since been found to be typical of most tape and foil applications (ref. 24).

The problem with the application of the aluminum foil tape developed in the prototype stage of the program. A 24-inch (0.6-m) wide tape was applied to the magnesium and aluminum panels to give them a one-piece aluminum appearance. During the prototype thermal-vacuum testing, the tape lifted from the surface on some of the panels, stretched and then collapsed into a long crease or wrinkle when the surface was returned to atmospheric pressure (see fig. 11). The panel temperatures were all below 100° F (40° C) during the test while the pressure was about 10⁻⁸ torr. This wrinkling or lifting condition was felt to produce too much uncertainty in heat exchange for a long-term mission. A program was undertaken to determine ways around this lifting problem. A preliminary investigation revealed that the use of a narrower tape was a more acceptable pro-

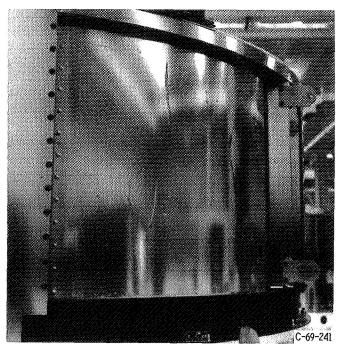


Figure 11. - Lifting of aluminum foil tape.

cedure. The subsequent work was then limited to 2- and 3-inch (0.05- and 0.08-m) tape widths.

Application Studies

This application technique study consisted of a literature survey, conferences with the manufacturer and other users, and an experimental program conducted at Lewis. It was suspected that the blistering was caused by the outgassing of the silicone adhesive. To test this hypothesis several tape samples 3 inches wide by 6 inches (0.08 by 0.15 m) long (with the liner removed) were placed in a vacuum oven and baked for 24 hours at 250° F (120° C). Shortly after the temperature elevation was started, deposits started to form on the cooler glass ports of the vacuum oven. This deposit continued to build up while the bakeout continued. Infrared absorbence analysis of this deposit revealed it to be a form of silicone from the adhesive.

The application technique experimental program consisted of a series of three tests. The first of these tests evaluated the effect of substrate condition. A piece of 2024 aluminum plate 12 by 24 by 0.06 inches $(0.3 \times 0.6 \times 0.002 \text{ m})$ thick was chosen as a test bed. The plate was detergent washed and then cleaned with xylene. Three strips of aluminum foil tape 3 inches (0.08 m) wide that had been baked out at 250° F $(120^{\circ}$ C)

for 3 hours were applied along with three strips of unbaked tape of the same size. When subjected to vacuum (less than 10^{-4} torr) at room temperature, the unbaked samples immediately developed small blisters. After 20 hours all samples developed blisters and the test was terminated.

The next test investigated tape application pressure. The 2024 aluminum substrate was vapor honed and then ultrasonically cleaned in Freon. Both unbaked tape and tape that had been vacuum baked for 24 hours at 250° F (120° C) were used in this test. The tape was applied in an overlapping pattern of three strips 3 inches (0.08 m) wide with approximately a 1/8-inch (0.003-m) overlap. The first of the three strips was applied with light pressure (about 3 lb (13 N) force on approx. 6 in. 2 (4×10⁻³ m²) or just enough to set the tape), the second with the normal pressure (about 6 lb (27 N) force), and the third with heavy pressure (about 10 lb (45 N) force). The test temperatures were room temperature, 100°, 125°, 150°, and 180° F (38°, 52°, 66°, and 82° C). The specimens were held at room temperature in vacuum of 10⁻⁸ torr for 72 hours. After this period, only the lightly applied, baked-out tape remained free of blisters. Holding the specimens at each of the other test temperatures for 24 hours resulted in increasing the size of the blisters on the tape. Only the one strip of lightly applied, vacuum-baked tape continued to exhibit good adhesion. It is believed that the light application pressure kept the adhesive from sealing the gas escape paths. On return to atmospheric conditions, the majority of the blistering disappeared and the tape looked exceptionally good. This points out the need to observe the samples under the test conditions.

The final test of this program investigated surface conditioning methods and perforations in the tape. Only unbaked tape was used in this test sequence. All the tape was applied with light application pressure. The test bed was the 2024 aluminum plate that was used throughout this program. The previous tape was stripped off and the plate was vapor honed and ultrasonically cleaned in Freon. Part of the surface was roughened with 60 grit paper with the grooves running perpendicular to the direction of tape application and then cleaned with trichlorethylene. Two layers of 2-inch (0.05-m) wide tape were applied in an overlapping pattern to this area. Another region was coated with a silicone primer and two overlapping layers of 2-inch- (0.05-m-) wide tape were applied to this region. A fourth region was cleaned with trichlorethylene and two layers of 2-inch (0.05-m) wide aluminum tape perforated with 1/32-inch- $(8\times10^{-4}$ -m-) diameter holes on 1/2-inch (0.015-m) centers were applied. The perforations were placed in the tape before the liner was removed. Finally, two layers of 2-inch-(0.05-m-) wide tape were applied as a control sample. The test was run in what had become the standard manner: 24 hours each at room temperature, 100° , 125° , 150° . and 180° F (38°, 52°, 66°, and 82° C) with the system held under vacuum at room temperatures over weekends. The results were that all samples except the perforated specimen were blistered within the first 20 hours at room temperature. Increasing the

temperatures did not cause the perforated tape to develop blisters. It did, however, cause an increase in size of the blisters in the other samples; the blisters appeared to grow together into a larger blister. Hence, perforated tape is as satisfactory at preventing blistering as the vacuum bakeout. Again, it is believed that the light application pressure was very important in that it kept the adhesive from flowing into the pinholes and thereby sealing them. Thus, the gas was given a good escape path and this prevented the development of blisters.

Evaluation

As a result of this program, two ways were found in which the tape could be applied with reasonable assurance that it would remain on the surface. The tape could either be vacuum baked at 250° F (120° C) for 24 hours or be perforated with 1/32-inch- (8×10^{-4} -m) diameter holes on 1/2-inch (0.015-m) centers. Since about 30 yards (27 m) of 2-inch- (0.05-m-) wide tape was involved in the thermal control pattern of the SERT II, it was decided that perforating the tape would be much more practical. Light application pressure must also be used.

The surfaces coated with the tape were subjected to thermal-vacuum testing to demonstrate that the perforated tape would remain on the surfaces without blistering. These surfaces were subjected to test temperatures of 140° and -40° F (60° and -40° C) for 24 hours each, then cycled between these limits 10 times, and then were held for

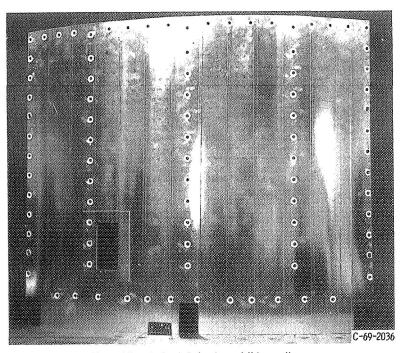


Figure 12. - Perforated aluminum foil tape pattern.

24 hours again at each temperature extreme, all the while maintaining a pressure below 10^{-7} torr. Figure 12 shows a typical panel of perforated tape after such a test. There were no discernible blisters observed either during the test or when the panel was removed. Care was taken to ensure that there was a row of perforations to outgas the overlap joint.

The optical properties were measured on small samples prepared at the same time as foil panels by the Cary 14 spectrophotometer, vacuum calorimeter, and Lion Optical Comparator. The results are shown in table II. Since the taped surfaces generally are not expected to see sunlight in space, no solar simulation test was conducted on these coatings.

Summary Remarks

The experimental investigation into the application of aluminum foil tape has revealed that the blistering or self-lifting of the tape is not solely a temperature dependent process, but is a pressure differential phenomenon. All the tape that blistered did so within the first day when the test plate was at room temperature and the pressure at 10^{-7} torr or lower. Close examination of the samples during pumpdown showed the development of slight wrinkling in areas that later developed blisters when the pressure reached about 10^{-4} torr.

The blistering was found to be alleviated by either baking the tape in vacuum at 250° F (120° C) for about 24 hours to remove the outgassing constituent of the adhesive or perforating the tape without the vacuum bakeout. This latter method was used on the SERT II project.

Regardless of the method used to prepare the tape, it was found that the tape had to be applied with light application pressure to prevent the adhesive from sealing the pin holes or other gas escape paths under the tape.

POLISHED ALUMINUM SURFACES

Background

Polished aluminum surfaces are used on some of the Sun facing panels of the vehicle (see fig. 2). The decision to use polished metal here was motivated by the change to aluminum substrates for the Z-93 white paint application. These panels were made of premium grade Alclad 2024 aluminum alloy which has a thin exterior coating of pure aluminum over the 2024 alloy. It was felt that it would be easy to polish the required area to a given surface finish and obtain reproducible optical properties.

The polishing of the aluminum surface was not difficult. In the following paragraphs a summary of the techniques employed will be presented.

Polishing Techniques

The panels were received for polishing after the interior sides had been painted black and after an ultrasonic cleaning in Freon. Those areas that were not to be polished were masked and then the panel was mounted for polishing on a curved wooden mandrel which matched the curvature of the panel.

The polishing of the panels was accomplished in two stages: a rough polishing operation and a final polishing operation. The rough polishing was accomplished with a 10-inch- (0.25-m-) diameter by 2-inch- (0.05-m-) wide Canton flannel buffing wheel using white jewelers rouge polishing compound. The buffed surface was cleaned by washing the panel with a cleanser and rinsing with clean, running water. The final polishing was accomplished with clean flannel buffing wheels only. The final polished surface was cleaned only by blowing air over the surface to remove any large particles. The wheels were mounted on a pneumatic drill motor. Care had to be taken not to cut through the thin, soft aluminum cladding, and also not to scratch the surface in cleaning.

Evaluation

The first test of the polished surface was an inspection for appearance. The finished polished surface was to have a uniform, mirror-like appearance without any deep scratches or visible blemishes.

The optical properties of 4-inch (0.1-m) specimens polished at the same time as the panels were determined by Cary 14 spectrophotometer measurements and by the Lion Optical Comparator (see table I). Solar simulation testing was run on polished aluminum surfaces along with the Z-93 white paint samples. The resulting temperatures from this test are shown in figure 10(a). The computed absorptance is shown in figure 10(b). The indicated decrease in computed absorptance with time is interpreted to be due to an increase in emittance with time rather than a real change in the absorptance. This change in emittance amounts to an increase of 0.006 in 400 hours which is not an excessive degradation. The resultant range of computed absorptance of 0.24 to 0.25 is in good agreement with the design range of 0.21 to 0.31.

The polished panels were subjected to the same thermal-vacuum testing as the Z-93 white paint coated panels. In this case the failure criteria was the clouding of the polished surface. This did not happen so no repolishing of these surfaces was required.

Summary Remarks

The polishing of the Alclad aluminum panels caused the least difficulty of any of the

SERT II coatings. The polishing was done with flannel buffs and white jewelers rouge. Care had to be taken so that the polishing operation did not cut through the thin, pure aluminum cladding over the alloy. With the proper care and diligence, uniformly polished surfaces with reproducible optical properties were attained.

CONCLUDING REMARKS

The coating application problems that have been encountered in this program have been solved. These problems developed in the prototype stage of the program even though the previous experimental program indicated that there would be no problems.

The application of the white inorganic paint (Z-93) was particularly discouraging since, for considerable time, there appeared to be no consistency in the results, and, hence, no way in which a logical application technique could be evolved. However, this application technique was eventually developed. The Z-93 paint could be satisfactorily applied using paint made from zinc oxide powder calcined at about 1200° F (650° C). It was found that a rub prime of the surface to be coated was an excellent way to ensure that the surface is prepared well enough to promote good paint adhesion. Furthermore, the paint adhesion was found to be influenced strongly by the environmental conditions which control the paint drying. A bakeout immediately after painting was used to overcome the adverse high humidity conditions. Using these techniques the Z-93 paint was successfully applied.

The aluminum foil tape blistering problem was solved in a more straightforward fashion. Of the possible solutions, the combination of a perforated pattern coupled with light application pressure was demonstrated to be acceptable for this vehicle. The polishing of the aluminum was accomplished with a minimum of difficulty.

The mechanical and optical properties of these coatings were ascertained by ground measurements. A system of periodic optical property measurements on samples was used to guard against using contaminated surfaces as flight hardware.

Lewis Research Center,
National Aeronautics and Space Administration,
Cleveland, Ohio, September 3, 1970,
704-00.

APPENDIX A

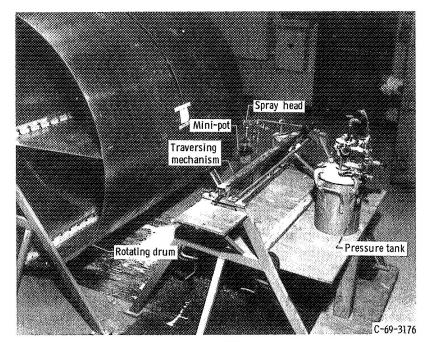
AUTOMATIC PAINT MACHINE

In an attempt to apply the Z-93 white paint uniformly to all of the panels, work was undertaken on the development of an automatic spray paint machine (see fig. 13(a)). The 5-foot (1.5-m) diameter drum held the six panels that made up a set. The spray head traversed horizontally back and forth while the drum rotated. The paint was placed in a pressure tank and force fed to the spray head. The paint was kept mixed in the tank by use of an agitator.

The first problem that arose was sputtering or uneven flow of paint from the spray head. This was solved by use of a mini-pot (or small suction tank) in the feed line below the spray head (see fig. 13(b)). The added mini-pot provided the spray gun with a constant source of fluid. The mini-pot traversed back and forth with the spray head. This added weight caused a slight vibration which kept the Z-93 paint in the mini-pot agitated.

This system did work and was used to paint a set of test panels. However, as the work with the machine was continued, problems with the paint texture developed. The pressure used on the paint pressure tank was a prime variable in determining the texture of the coating. This effect would vary day by day, so that it was difficult to know what setting to use for a given paint run. Another problem was the uneven drying of the paint. Parts of a panel would dry to a rough texture and parts would dry to a glossy texture. Baking the paint with a lamp bank after painting did not cure this problem.

The Z-93 paint application procedure was found to require day-by-day variations to account for changes in local conditions. Hence, it was advisable to make trial paint runs before painting a set of panels. With the paint machine it took 2 to 3 days to prepare all of the panels for a paint run. This made it impossible to make the trial runs before the actual paint run. While these problems were being studied, the painter developed his hand technique to a point where he could apply a uniform coating. So, work with the machine was terminated.



(a) Overall view.

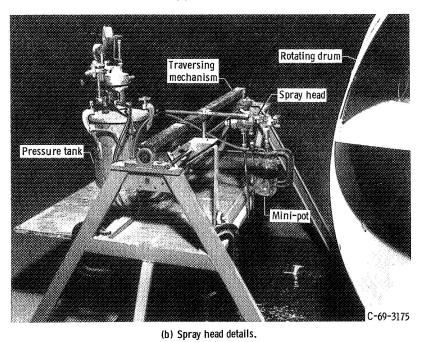


Figure 13. - Z-93 automatic paint machine.

APPENDIX B

7-93 WHITE INORGANIC PAINT PROCESS SPECIFICATION

This specification establishes the preparation, application, and testing techniques required to apply Z-93 white inorganic coatings to the SERT II vehicle.

GENERAL REQUIREMENTS

Environmental Conditions

For any paint application the room temperature shall be $75^{\circ}\pm10^{\circ}$ F ($24^{\circ}\pm6^{\circ}$ C) and the relative humidity shall be 55 ± 25 percent.

Equipment

Spray guns and other accessories shall be kept clean and shall not be used for applying other coatings. Rinse and flush thoroughly with distilled or deionized water before and after each operation.

Handling

Parts, both prior to and after coating, shall be protected from dust, finger prints, oil, etc. Operator shall wear clean, lint-free gloves and use clean tools at all times during handling.

Surface Protection

The cured Z-93 white painted surface can be covered only by clear teflon or clean kraft paper.

SURFACE PREPARATION

Masking

All areas of the panels that are not to be painted are to be masked. Use clean masking tape such as Mystik no. 7300 and teflon or polyethylene film to cover these areas. Seal the panel holes on the back side with masking tape.

Sanding

Sand all areas to be painted with no. 220 grit aluminum oxide paper. Wet sanding is to be employed. All panel edges that are to be painted shall also be sanded. Wash off the residue with distilled water.

Cleaning

Scour the sanded surfaces with a commercial scouring powder, such as trisodium phosphate. Rinse thoroughly with distilled water. Then, clean entire surface including edges with detergent such as Alconox. Rinse thoroughly with distilled water. Test surfaces for chemical cleanliness by flowing distilled water over the entire area. If no water breaks occur, proceed to the next step. If water breaks do occur, continue cleaning until the surface passes this test.

Rub Priming

All areas to be painted including edges are to be primed in the following manner:

- (1) Z-93 white inorganic paint is to be rubbed into the surfaces to be painted using cheesecloth or nylon cloth. The cloth is to be changed if it becomes dirty. The coating is to be rubbed into the metal and should result in a thin coating giving a dull grey appearance.
- (2) After the rub prime surface is completely dry, test for adhesion by wetting the primed surface with distilled water and rubbing gently with a clean, wet cheesecloth pad. If the prime coat is adhering well, it will not come off. If any of the prime coat does come off, sand that area again, rinse and rub prime again. Continue this procedure until the entire area passes this rub prime adhesion test.

Processing Schedule

All parts to be painted must be processed through the rub prime stage before stopping. Cleaned panels must not be left overnight.

PAINT PREPARATION

Coating Composition

The paint shall consist of a mixture of zinc oxide powder (New Jersey Zinc Co., no. SP-500), potassium silicate (Sylvanic Electric Products, Inc., no. PS-7), and distilled water. The zinc oxide shall be calcined or heat treated at $1200^{\circ}\pm25^{\circ}$ F ($650^{\circ}\pm15^{\circ}$ C) for $16\pm1/2$ hours in an air furnace before compounding the paint. The paint shall be mixed according to table III.

| | Basic fo | ormula | Typical SERT II | | | |
|--------------------|----------|--------|-----------------|------|---------------|--|
| | g | ml | mix | | , | |
| | ь | | lb | g | ml | |
| Zinc oxide | 200 | | 3.51 | 1600 | | |
| Potassium silicate | | 100 | 2.33 | | 800 | |
| Distilled water | | 100 | 1.75 | | 800 | |

TABLE III. - Z-93 PAINT COMPOSITION

Coating Preparation

<u>Milling</u>. - All jars and balls must be clean before starting the paint mixing. Into a dense alumina jar (9-in. (0.3-m) diam.) place the paint ingredients and 3.4 pounds (1544 gm) of 1/2-inch- (0.015-m-) diameter alumina balls as the grinding media (for the typical SERT II mix). Ball mill the mixture at 100 rpm for $1\frac{1}{2}\pm1/2$ hours.

<u>Viscosity</u>. - Check the viscosity of the resulting paint with the Thomas Stormer Viscosimeter. If the viscosity is more than 68 Krebs units, add distilled water and ball mill again for 5 to 10 minutes. Repeat procedure until the viscosity is between 65 and 68 Krebs units.

<u>Filter</u>. - Filter the mill charge through a nylon stocking (approx. 80 mesh) into a clean glass container. Do not use a metal filter.

Shelf life. - Use the paint the same day that it is mixed.

COATING APPLICATION

Equipment

The spray equipment shall be pressurized with nitrogen only. Satisfactory coatings have been applied using a Binks Model 62 spray gun with a pressure pot and a 66 PE air nozzle, 63 B fluid nozzle and B63A needle. Other model guns and nozzles can be used with equal success. Typical pressure settings are 20 psi $(1.38\times10^5~\text{N/m}^2)$ line pressure with 3 to 5 psi $(2.1\times10^4~\text{to }3.4\times10^4~\text{N/m}^2)$ on pot. The gun is to be adjusted to obtain uniform passes and minimum overspray. During the spraying operation, periodic swirling of the paint pot is necessary to prevent pigment settling.

Paint Procedure

First coat:

- (1) Mist coat the rub primed surface with distilled water if the rub prime coat has been allowed to dry. Wait until the water gloss has disappeared before proceeding.
- (2) Fog a light coat of Z-93 paint onto the rub primed surface with the gun held perpendicular to the surface and about 8 to 12 inches (0.2 to 0.3 m) from the surface. When the gloss from this coat disappears, apply a full coat of two wet passes, one perpendicular to the other, to obtain a $2\frac{1}{2}\pm1$ mil (6.5×10⁻⁴±2.5×10⁻⁴ m) dry film thickness. Paint the panel edges also. Wait between passes until the gloss from the first disappears.
- (3) The spray gun should move in such a manner that the coating material will wet the surface evenly without runs or sags. The coating material must be applied as a continuous wet film.
- (4) In humid weather (at the high end of the permitted relative humidity), the paint tends to be thin and when applied will not dry to the desired rough texture. Under these conditions, the panel must be subjected to heating immediately after the first full coat application. Hold the panels under the heat lamp bank until the steam stops rising from the surface. At this point, the desired rough surface texture should be evident.
- (5) Allow coating to air dry at room temperature for 1 to 4 hours. If the panel has been heated to drive off moisture, do not paint this panel again until it is cool. Final coat:
- (1) Mist coat all Z-93 white painted surfaces with distilled water and wait until the water gloss disappears before continuing.
- (2) Apply final coat in the same manner as described above, adjusting the number of wet passes so that the final dry film thickness will be 5.5±1.5 mils (1.4×10⁻⁴± 0.4×10⁻⁴ m).

Note that the paint will build up faster on this second coating. Care must be taken not to apply too thick of a coating. If necessary, additional coats may be applied after the air cure when thickness is checked. Mist coat with distilled water before applying wet coats of paint.

Curing:

- (1) Air dry the coatings at room temperature for a period of 12 to 24 hours before demasking panels.
 - (2) Oven bake painted parts according to the following schedule:
 - (a) Raise temperature to 150° F (65° C) and maintain for 1 hour.
- (b) Raise temperature from 150° F (65° C) to $270^{\circ} \pm 20^{\circ}$ F ($130^{\circ} \pm 11^{\circ}$ C) in about 20 minutes and hold panels at this high temperature for 2 hours.
 - (c) Remove panels from oven and air cure to room temperature.
 - (3) Clean out all mounting holes by hand.

QUALITY ASSURANCE

Appearance

When visually examined, the Z-93 coating shall appear flat white and shall be free of blisters, pin-holes, runs, cracks, or any other defects which could adversely effect the proper function of the coating.

Film Thickness

Thickness readings shall be made on the coated panel. The measurements shall be made with an eddy current, dry film thickness gage such as Twin Cities gage. The dry film thickness shall be 5.5 ± 1.5 mils $(1.4\times10^{-4}\pm0.4\times10^{-4})$ m).

Testing

Test specimens shall be processed concurrently with the parts they represent using the same procedures. Two 1 by 1 inch (0.025 by 0.025 m) specimens and four 4 by 4 by 0.040 inch (0.1 by 0.1 by 0.001 m) thick Alclad aluminum specimens must be prepared. On the 4-inch- (0.1-m-) square samples only 4 by $2\frac{1}{2}$ inches (0.1 by 0.06 m) of the surface has to be painted. The small samples will be used for the vacuum calorimetric optical property measurements while the larger samples will be used for bend

adhesion, thermal shock, and Cary 14 spectrophotometer measurements. Bend adhesion test:

One of the coated 4 by $2\frac{1}{2}$ inch (0. 1 by 0.06 m) painted Alclad samples shall be subjected to the conical mandrel bend test described in Federal Test Method Standard 141, Method 6222. The short dimension of the sample is placed over the mandrel at $3/4\pm1/16$ inch (0.02±1.6×10⁻³ m) from the base. After bending, a piece of 3M Company no. 250 masking tape 1 inch (0.025 m) wide is firmly applied along the entire length of the convex surface of the bend, then abruptly pulled off.

(2) The coating on the tested area shall not show a loss of adhesion in excess of 20 percent. Adhesion failure within 1/8 inch $(3.2 \times 10^{-3} \text{ m})$ of the panel edges shall be discounted.

Thermal-shock stability:

- (1) One of the coated 4 by 2.5 by 0.040 inch (0.1 by 0.06 by 0.001 m) Alclad samples shall be heated to $490^{\circ}\pm10^{\circ}$ F ($255^{\circ}\pm5^{\circ}$ C), immersed immediately after removal from the oven into liquid nitrogen until the temperature is stabilized, and then removed from the liquid nitrogen and allowed to reach room temperature. Two such cycles shall be performed.
- (2) After the test is complete, the sample shall not show a loss of adhesion in excess of 10 percent.

Optical property measurements:

(1) The Z-93 white paint coating on the test samples shall have the following room temperature properties:

$$\epsilon$$
 = 0.90±0.05 (from both Lion Optical Comparator and vacuum calorimetric measurements)
$$\alpha_{\rm S} = 0.16\pm0.03 \qquad {\rm (Cary~14~spectrophotometric~measurement)}$$

$$\alpha_{\rm S} = 0.19\pm0.03 \qquad {\rm (Lion~Optical~Comparator~measurement)}$$

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